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THE ELECTRICAL CONDUCTIVITY OF COPPER OXIDE

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THE ELECTRICAL CONDUCTIVITY OF COPPER OXIDE

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ABSTRACT: The temperature dependency of electrical conductivity of copper oxide plates with different oxygen contents will be investigated. The electrical conductivity of pure copper oxide at 12° is $2.7 \cdot 10^{-10} \text{ ohm}^{-1} \text{ cm}^{-1}$.

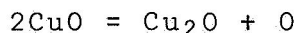
The dissociation energy of the electrons in Cu_2O amounts to 0.72 volts. The electrical conductivity of copper oxide increases rapidly with the oxygen content; the latter is not larger than 0.1% of the weight. The dissociation work of electrons in well-conducted tests of copper oxide varies from 0.134 to 0.129 volts.

Electrical properties of copper oxide in recent times have formed the basis of a large number of experimental investigations owing to the great technical significance of Cu_2O and because it is easy to prepare copper oxide samples in a convenient form for experiment. /453*

The published works emphasize the essential role of excess oxygen in the electrical conductivity of copper oxide.

Auwers and Kerschbaum [1] found a significant decrease in electrical conductivity of copper oxide plates after heating the latter for many hours under high vacuum or in an atmosphere of nitrogen. LeBlanc and Sachse [2] further investigated the electrical conductivity of copper oxide powder in connection with the working conditions which altered the content of excess oxygen in Cu_2O . The electrical conductivity of copper oxide powder heated in vacuum in which there were no detectable traces of oxygen, amounted to $10^{-9} \Omega^{-1} \text{ cm}^{-1}$ at 20° C and increased to $10^{-6} \Omega^{-1} \text{ cm}^{-1}$, since the Cu_2O in the air absorbed less than one per mil of oxygen. The same author reached a similar conclusion in other experiments with nickel and cobalt oxide [3]. This excess oxygen which they gave the name "active oxygen", exists in the opinion of the author in a quasi dissolved state. They consider the possibility of its existence in the form of cupric oxide improbable, because it is very easy to separate the oxygen from copper oxide powder at temperatures which correspond to an insufficient Cu_2O dissociation work according to the equation /454

*Numbers in the margin indicate pagination in the foreign text.



Dubar [4] obtained similar results in investigation of copper oxide plates, in which he changed the conditions of the thermal treatment. A thorough investigation of these conditions which was carried out by Waibel [5], has shown the possibility of significant increase of electrical conductivity of copper oxide through heating the test material in air at different temperatures and a subsequently rapid cooling. The sense of these operations remained, however, quite incomprehensible. All considerations of the influence of excess oxygen on the electrical conductivity of copper oxide, which have been put forth in the reference works, are of a purely qualitative nature. The dissertation of Vogt [6] which had as its task the investigation of the electrical characteristics of copper oxide as a typical electron semiconductor, is by no means a full treatment of the phenomenon and was carried out only on samples with a single oxygen content.

In the present work we have set ourselves the task of investigating the electrical conductivity of copper oxide dependent on the excess oxygen content, and to explain the role of the oxygen content as the chief factor which the abrupt fluctuation of the electrical conductivity of copper oxide in as wide a range as $10^{-2} - 10^{-9} \Omega^{-1} \text{ cm}^{-1}$ requires. /455

Moreover, we have set ourselves the task of examining the question of the specific electrical conductivity of copper oxide. The already obtained results which have been published by various authors and which were based on very poorly conductive samples, provide little reason to believe that the measured electrical conductivity is limited by the oxygen remaining behind in the CuO . Such considerations are all the more inappropriate, since many scholars are of the opinion that the electrical conductivity of semiconductors will develop only through the impurities present in them. In a purer form such a semiconductor should appear as an insulator.

According to the ideas of A.F. Joffé, one can resolve this question by measuring the temperature range of electrical conductivity of semiconductors with different impurity contents, from which it is to be expected that the temperature dependency curves obtained at high temperatures will fall together in a single curve corresponding the specific electrical conductivity of copper oxide because the electron concentrations required by impurities will be small under such conditions in comparison with the conductivity brought about by the dissociation of copper oxide.

Moreover, the thorough investigation of a material which, according to its state, possesses various electrical conductivities, promises new experimental material for the understanding of the mechanism of conductivity in semiconductors.

As starting material for the preparation of Cu_2O , an old supply of very pure electrolytic copper in the form of a 0.35 mm thick band which was prepared on special order by the "Sewkabel" factory was used. The copper oxide was produced as usual by a one-hour heating of metallic copper in an electrical oven at a temperature of 1030°C . The cooling of the prepared Cu_2O samples was carried out in water vapor in such a way that the sample - direct from the oven - was placed in a tube through which water vapor was passed. The clear, dark-red copper oxide thus prepared was employed as the starting material for further treatment. /456

Upon microscopic examination of such samples in light passing through them with a magnification of 500 X, one saw in addition to dark-red cavities also black needle-shaped crystals grown together usually occurring in pairs in a V-form. These crystals, which have already been observed by Dubar, represent cupric oxide crystals which, as Dubar has noted disappear after heat treatment which separates the excess oxygen. In our experiments this was done by heating the plates in high vacuum. The crystals also disappear, we ascertained, when one heats a plate to 1000° and immediately cools it by chilling it in cold water. This occurrence of CuO crystals in the latter case is explained apparently by the fact that the excess oxygen found in the glowing copper has no time to separate into discrete crystals and remains in molecular solution. The samples with increased conductivity were prepared by us according to the Waiber method, that is, through a second heating of the above-described starting copper oxide plates six hours long at various temperatures with subsequent cooling in cold water. The outer layer of cupric oxide was separated by treatment with 10% hydrochloric acid followed by a 10% solution of ammonia.

As the microscopic analysis has shown, needle-like copper oxide crystals are absent from such plates, which the investigation of the real dependency of electrical conductivity of oxygen content has shown. It is evident that highly conductive microscopic crystals of CuO which practically do not alter the amount of electrical conductivity contribute to the total content of excess oxygen which is determined through the analysis. The copper oxide samples which contain practically no excess oxygen, were produced by a 6-7 hour heating of the starting material in vacuum of 10^{-4} to 10^{-5} mm mercury at 800°C . The samples were suspended in a quartz tube surrounded by tungsten wire. This oven, which, in turn, was located in a wide quartz tube surrounded by a copper band, was arranged in a glass vessel fitted with a ground glass top; the top of the vessel led to a vacuum pump. The samples produced, as the analyses and the conductivity measurements have shown, are gradually charged with oxygen while they are in the air. /457

For the determination of excess oxygen content in Cu_2O , a method of direct analysis was worked out. A comparison of the excess oxygen content in recently published works shows a great discrepancy in results. Vogt [6] gives the value for CuO content in

Cu₂O as 3%; Dubar, 1.5%; according to Sachse [2] Cu₂O contains 0.6 atoms per mil of oxygen, which corresponds to 0.067% CuO. The first two authors determined the O content by an indirect method, in which they determined the total Cu content in Cu₂O. The method of analysis which Sachse employed was not given by him.

We determined the excess oxygen content in Cu₂O according to the method which is similar to the iodometric method Sachse employed for the analysis of nickel oxide. This method consists of the dissolution of Cu₂O in an acid KI solution. The Cu₂O is thus converted to CuI, and the excess oxygen oxidizes the hydroiodic acid with the simultaneous release of an equivalent amount of free iodine, which is titrated against a hyposulfite solution. In the event that the oxygen in the plate exists in the form of CuO, we obtain the same results: the cupric oxide, upon solution is converted into CuI₂ and the latter immediately decomposes according to the equation $CuI_2 = CuI + I$.¹ /458

¹The method contained two sources of error: first the oxidation of the KI acid solution by the oxygen of the air and the dissolved oxygen, which leads to an increased oxygen content in Cu₂O, and second, the adsorption of the liberated I by CuI, by which the amount of determinable oxygen can appear two times smaller. After many experiments, the following method was worked out which eliminates both sources of error. A quantity of Cu₂O of approximately 0.25 - 0.30 grams rubbed well in a mortar was introduced into a round-bottomed long-necked 60 cc flask; the neck of this flask was fitted with a rubber stopper in which a three-way stopcock for the introduction of CO₂ and a funnel with stopcocks were placed. 20 cc of boiled distilled water were placed in the flask and the powder was boiled for several minutes to remove the air bubbles; the flask was then filled with oxygen-free CO₂ with which it was washed well. After this, 40 cc of concentrated air-free KI acid solution were let through the funnel (the KI was free from iodine and potassium iodate); the Cu₂O powder was released completely in 2-3 minutes without liberation of CuI, owing to the large excess of KI; after this the solution was titrated first against a 1/50 Normal solution of hyposulfite to the disappearance of yellow, a color produced by free iodine, and was then titrated with a 1/50 N solution of I (with the addition of a starch solution) to a stable rose color. The titration did not end owing to the delay of the reaction, for which reason the accuracy of the analysis was about 10%. The above-noted solution of KI was prepared in the following manner: a solution of 30 grams KI in 120 cc water was reduced before the analysis in a retort fitted with a ground stopper to half of its volume; after this 10 cc of 12% HCl were added and the solution was again boiled for a short time to completely separate the dissolved oxygen and to decompose the traces of potassium iodate. The solution was then cooled in an atmosphere of CO₂ that titrated the least amount of liberated iodine against hyposulfite without addition of starch, and was quickly transferred to a graduated cylinder; 4/5 of the solution was poured through a funnel fitted with a stopcock and was taken for (Footnote continued next page).

The analytical results of Cu_2O plates whose electrical conductivity was investigated are given in Table 1.

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TABLE 1

Preparation Method		Oxygen Content in % wt.	Oxygen Content in atomic weight	Oxygen Content of CuO in % wt.
I	Heated in vacuum	not determinable		
III	Starting material	0.053	0.47	0.53
IV	A plate which was heated in vacuum after which, having remained 3 months in air, it was charged with oxygen	0.043	0.38	0.43
V	Heated 1 hour at 1000°C and cooled in water	0.060	0.53	0.60
VI	Heated 6 hours at 700°C and cooled in water	0.084	0.75	0.84
VII	Heated 7 hours at 600°C and cooled in water	0.090	0.80	0.90
VIII	Heated 6 hours at 500°C and cooled in water	0.101	0.89	1.01

As can be seen from Table 1 the heat treatment of Cu_2O which has been proposed by Waibel presents the possibility of increasing the excess oxygen content by only two times. The samples which were cooled from 1000°C , contain only somewhat more oxygen than the starting material, which one can explain by the diffusion of oxygen from the latter upon comparatively long cooling. The values obtained for the oxygen content are apparently closely connected to the preparation conditions of the Cu_2O and the starting material, and should not be referred to the Cu_2O samples which were prepared under other conditions.

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All of the samples whose analysis is given in Table 1, were subjected to conductivity measurements at different temperatures from 15 to 750°C . The measurements were carried out with a galvanometer having a sensitivity of $1.3 \cdot 10^{-10}$ Amp per scale section. The Cu_2O plates with the metal electrodes attached to both sides by the vacuum evaporation (around an electrode a basket was located) were squeezed in special forceps and placed in the electric ovens.

The temperature was measured by means of a thermal element whose junction was located under the sample. A portion of the measurements was controlled by the method of Vogt [6]. All of the

¹the analysis of Cu_2O ; 1/5 remained in the graduated cylinder for control purposes and was again titrated with an indicator.

investigated samples were tested in the absence of rectifying action, and assumed a volt ampere characteristic ranging from 0.1 to 10 volts, which in all cases yielded a line passing through the zero coordinate point. This circumstance points to the fact that at high temperatures, Cu_2O possesses electron conductivity. In Table 2 are given the results of conductivity measurements for the various Cu_2O samples described in Table 1; the numbering of the samples is the same. The same results are shown graphically in Figure 1 where also two curves (II, IX) corresponding to the electrical conductivity of pure copper oxide and cupric oxide in the form of pressed powders are given according to the data of Sachse and LeBlanc [2].

In Figure 1 the natural logarithms of the electrical conductivity are plotted on the ordinate axis, and on the abscissa the values $1/T$, where T is the absolute temperature. These coordinates are chosen according to the formula which the temperature dependency of the

$$\sigma = Ae^{-\frac{u}{kT}} \quad (1)$$

which the temperature dependency of the conductivity of a semiconductor represents.

TABLE 2

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I		III		IV		V		VI		VII		VIII	
t°C	σ	t°C	σ	t°C	σ	t°C	σ	t°C	σ	t°C	σ	t°C	σ
12°	$2.7 \cdot 10^{-10}$	0°	$1.17 \cdot 10^{-8}$	20°	$1.22 \cdot 10^{-8}$	0°	$1.0 \cdot 10^{-4}$	12°	$3.95 \cdot 10^{-4}$	0°	$3.6 \cdot 10^{-4}$	20°	$2.1 \cdot 10^{-3}$
110	$6.25 \cdot 10^{-8}$	20	$1.85 \cdot 10^{-8}$	60	$2.6 \cdot 10^{-8}$	20	$1.54 \cdot 10^{-4}$	20	$4.48 \cdot 10^{-4}$	17	$5.45 \cdot 10^{-4}$	50	$3.1 \cdot 10^{-3}$
145	$3.7 \cdot 10^{-7}$	50	$3.8 \cdot 10^{-8}$	120	$8.0 \cdot 10^{-8}$	70	$3.3 \cdot 10^{-4}$	60	$8.36 \cdot 10^{-4}$	60	$8.60 \cdot 10^{-4}$	100	$5.8 \cdot 10^{-3}$
160	$6.8 \cdot 10^{-7}$	115	$1.4 \cdot 10^{-8}$	160	$1.08 \cdot 10^{-4}$	120	$6.1 \cdot 10^{-4}$	75	$1.07 \cdot 10^{-3}$	120	$1.75 \cdot 10^{-3}$	200	$1.16 \cdot 10^{-2}$
227	$1.05 \cdot 10^{-5}$	165	$2.8 \cdot 10^{-8}$	220	$1.43 \cdot 10^{-4}$	207	$1.22 \cdot 10^{-3}$	140	$2.15 \cdot 10^{-3}$	180	$3.01 \cdot 10^{-3}$	300	$2.70 \cdot 10^{-2}$
270	$3.0 \cdot 10^{-5}$	259	$7.0 \cdot 10^{-8}$	270	$2.04 \cdot 10^{-4}$	270	$1.65 \cdot 10^{-3}$	170	$2.5 \cdot 10^{-3}$	264	$4.4 \cdot 10^{-3}$	400	$4.1 \cdot 10^{-2}$
315	$9.9 \cdot 10^{-5}$	302	$1.26 \cdot 10^{-4}$	320	$4.0 \cdot 10^{-4}$	360	$2.6 \cdot 10^{-3}$	227	$4.0 \cdot 10^{-3}$	350	$6.80 \cdot 10^{-3}$	510	$5.0 \cdot 10^{-2}$
350	$2.95 \cdot 10^{-4}$	353	$2.7 \cdot 10^{-4}$	420	$3.22 \cdot 10^{-3}$	420	$3.15 \cdot 10^{-3}$	256	$4.70 \cdot 10^{-3}$			635	$7.42 \cdot 10^{-2}$
395	$6.36 \cdot 10^{-4}$	407	$7.0 \cdot 10^{-4}$	470	$8.0 \cdot 10^{-3}$	480	$4.25 \cdot 10^{-3}$	311	$6.1 \cdot 10^{-3}$				
420	$1.16 \cdot 10^{-3}$	441	$1.95 \cdot 10^{-3}$	520	$1.3 \cdot 10^{-2}$	570	$7.5 \cdot 10^{-3}$	370	$8.2 \cdot 10^{-3}$				
470	$2.27 \cdot 10^{-3}$	484	$3.3 \cdot 10^{-3}$	560	$1.56 \cdot 10^{-2}$	640	$1.65 \cdot 10^{-2}$	393	$8.9 \cdot 10^{-3}$				
508	$3.84 \cdot 10^{-3}$	533	$6.32 \cdot 10^{-3}$	580	$1.88 \cdot 10^{-2}$	700	$3.6 \cdot 10^{-2}$						
570	$9.72 \cdot 10^{-3}$	590	$1.34 \cdot 10^{-2}$			780	$9 \cdot 10^{-2}$						
645	$2.40 \cdot 10^{-2}$												

As can be seen from Figure 1, the regularity required by Formula (1) for pure copper oxide in the whole investigated temperature range (Curve I) is sufficiently fulfilled.

The Curves III, IV, V, VI, VII, VIII, which correspond to the Cu_2O samples with increasing oxygen content, are in agreement when superposed; that is, the electrical conductivity of the Cu_2O samples at any temperature increases with the addition of oxygen. In apparent contradiction to this conclusion is the position of the curve corresponding to the starting material III, because Sample III contains more oxygen than IV. This anomaly is explained through the data from the microscopic analysis, through which the crystals in Sample III of CuO will be found. A conductivity of Sample VII deviating somewhat from the total picture is apparently connected with accidental temperature fluctuation during its preparation.

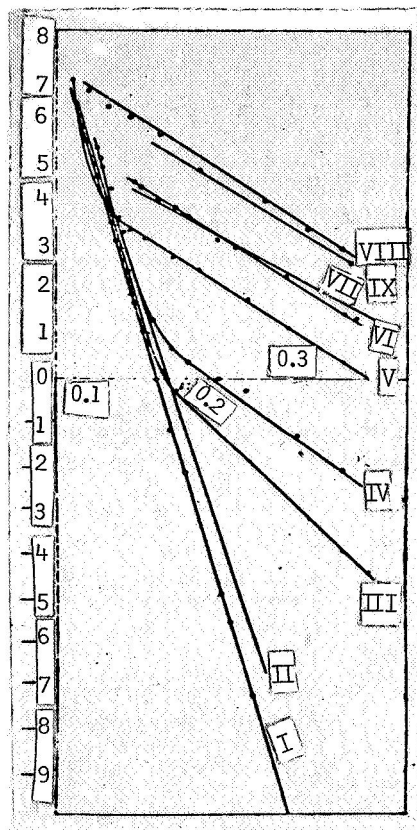


Fig. 1.

Abcissa: $\frac{1}{T \cdot 10^{-2}}$

Ordinate: $\ln(\sigma \cdot 10^4)$.

The original linear dependence of $\ln \sigma$ on T disappears in the range of higher temperatures, and the curves asymptotically approach the curve of pure Cu_2O but do not intersect it.

The falling together of the curves occurs at a higher temperatures, the larger the impurity content in the sample is. The total section for all curves must in agreement with the opinion stated at the outset of this paper correspond to the specific electrical conductivity of Cu_2O since this section of the curve within the limits of accuracy of our experiment lies on Line I for pure Cu_2O , one has sufficient grounds to assume that this line represents the specific conductivity of copper oxide also at the lower temperature range; that is, in

other words, the electrical conductivity, which is required only by the heat dissociation of Cu_2O . The conductivity values obtained by us correspond at least to the order of magnitude of pure copper oxide.

We are now going to consider the conductivity action in relation to the impurity content.

When we compare Curves VIII and IX with one another, we see that the absolute magnitude of the electrical conductivity of the

CuO samples with the highest oxygen contents are higher than the conductivity of pure cupric oxide powder. Thus σ for CuO at room temperature amounts to $6.4 \cdot 10^{-4} \Omega^{-1} \text{ cm}^{-1}$ and for Sample VIII, $3.5 \cdot 10^{-3} \Omega^{-1} \text{ cm}^{-1}$; Waibel [5] succeeded in producing samples of CuO with the electrical conductivity of the order of magnitude $1.4 \cdot 10^{-2} \Omega^{-1} \text{ cm}^{-1}$.²

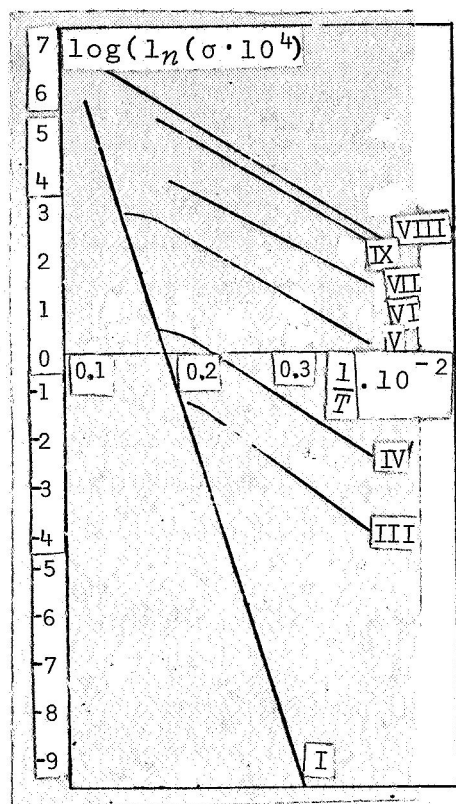


Fig. 2.

The slope value corresponds to the magnitude u/k in Formula (1) from which it is easy to calculate the magnitude u for the dissociation work of the electrons. These values are given in the second half of Table 3.

The results obtained by us obtain clear physical significance after the following conversions: When one subtracts the electrical conductivity of pure Cu₂O from the absolute value of the electrical conductivity of a sample with oxygen impurities at the same temperature, one obtains the electrical conductivity which is engendered by the impurity. When one carries out such a conversion along the whole curve, one obtains a curve which represents the temperature range of this conductivity. The results of a similar conversion are given in Figure 2.

From Figure 2 it can be seen that the slopes of the curve fall together with sufficient accuracy with the slope of the curve for pure CuO, with the exception of Sample III which deviates from the others owing to the already noted lack of homogeneity. In Table 3 the numerical values of the curves for pure copper oxide, pure cupric oxide and also for all converted curves are given.

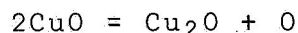
²Note on Revision: In a work appearing in the meantime of P. Guillery (Ann. d. Phys. Vol. 14, No. 5, pp. 216-220, 1932) a significantly higher value has been found for the electrical conductivity of cupric oxide in a compact form (order of magnitude $1 \text{ ohm}^{-1} \text{ cm}^{-1}$). From this it can be concluded that the results of LeBlanc and Sachse [2] for copper oxide powder were heavily influenced by filter resistance. This must be taken into consideration in the comparison of the electron release work calculated for the cupric oxide powder and for the cooled compressed copper oxide samples investigated by us.

TABLE 3

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Nos.	I	II	III	IV	V	VI	VII	VIII	IX
Slope	8100	7320	2170	1720	1560	1520	1500	1560	1600
Dissociation Work of the Electron in Volts	0,723	0,618	0,187	0,148	0,131	0,131	0,129	0,134	0,138
Constant A	44,2	—	0,0012	0,0060	0,031	0,080	0,077	0,404	0,385

The results received permit the assumption that the excess oxygen is contained in Cu_2O in the form of a molecular solution of CuO . For such solution of CuO in the lattice of Cu_2O the stability conditions of the excess oxygen atoms, even if chemically bound, are apparently quite different than the stability conditions in the lattice of pure cupric oxide. In the first case the heat dissociation of CuO should be begin according to the equation

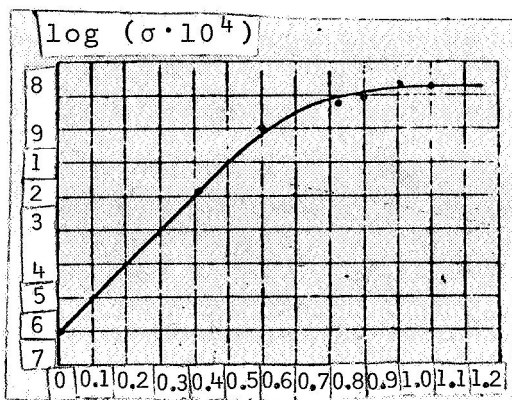


at lower temperatures than in the case of pure CuO ; for this reason it seems to us that the above-noted considerations of LeBlanc and Sachse [2] are not convincing.

In addition to the constant u , Equation (1) contains still another constant A . The value of $\log A$ can be shown graphically by extension of the line in Figure 2 to the point of intersection with the ordinate ($T = \infty$). These results are given in Table 3.

When we compare the numerical values of electrical conductivity at room temperature for a various Cu_2O samples, we note that this conductivity becomes equal to the conductivity of pure cupric oxide as soon as the cupric oxide content becomes as great as 0.6%, and it changes little upon further increase of the oxygen content.

The graphic presentation of this dependency is given in Figure 3; the \ln of the electrical conductivity at room temperature are



CuO Content in % Wt.

Fig. 3.

plotted on the ordinate axis, and on the abscissa the per cent copper oxide. The curve range is apparently connected with the mechanism of electron motion in a Cu_2O lattice, in which the impurity molecules are included; an investigation of a similar system provides clarification of this mechanism, in which one, however, can study the impurity content in wider ranges than in the cases investigated by us and can explore, besides the electrical conductivity, still other physical properties of semiconductors. Such investigations are now being carried out in the Physical Techno-

logical Institute at Leningrad.

CONCLUSIONS

1. The electrical conductivity and temperature range of copper oxide samples which was produced by the oxidation of electrolytic copper plates and subjected to various heat treatments, were investigated.

2. An iodometric method for the direct analysis of oxygen content in copper oxide was worked out.

3. It was found that the electrical dependency of copper oxide increases with the increase of the oxygen content; and in a Cu_2O sample with 0.1% oxygen content by weight, the value of $2 \cdot 10^{-3} \Omega \text{cm}^{-1}$ at 20°C was obtained.

4. It is shown from the temperature range of the electrical conductivity for all samples that the conductivity of a Cu_2O sample heated in vacuum corresponds to the specific electrical conductivity of Cu_2O . /467

5. The electrical conductivity of pure copper oxide at 12°C amounts to $2.7 \cdot 10^{-10} \Omega^{-1} \text{cm}^{-1}$. The dissociation work calculated from the temperature range of electrical conductivity for one electron amounts to 0.72 volts.

6. The dissociation work of the electron in highly conductive samples is 0.134 to 0.129 volts, which corresponds to the dissociation work of the electrons in pure cupric oxide powder; according to the conclusions of Sachse this amounts to 0.138 volts.

7. The conjecture is expressed that the excess oxygen in Cu_2O is held in the form of a molecular solution of CuO .

8. The dependency of Cu_2O conductivity on the excess oxygen content is expressed through a curve which has the characteristics of a saturation curve.

In conclusion we owe our thanks to A.F. Joffé for his encouragement of this work and for his valuable advice while it was being carried out.

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Leningrad, June 1932.

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